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18JUL03 E823609-5 D02944
P01/7700 0.00-0316857.2

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Request for grant of a patent

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The Patent Office

Cardiff Road
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1. Your reference — SMC 60603/GB/P1

2. Patent application number *(The Patent Office will fill in this part)* — 0316857.2 — 18 JUL 2003

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

Avecia Limited
Hexagon House
Blackley
Manchester, M9 8ZS
United Kingdom

Patents ADP number *(if you know it)* — 07764137001

If the applicant is a corporate body, give the country/state of its incorporation — GB

4. Title of the invention — COMPOSITIONS

5. Name of your agent *(if you have one)*

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

~~FAWKES, David Melville~~

~~Avecia Limited~~
~~Hexagon House~~
~~Blackley~~
~~Manchester, M9 8ZS~~
~~United Kingdom~~

D Young + Co
21 Newfetter Lane
London
EC4A 1DA

Patents ADP number *(if you know it)* — ~~2180511002~~ 59006 ~~07764137001~~

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and *(if you know it)* the or each application number

Country	Priority application number *(if you know it)*	Date of filing *(day / month / year)*

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application	Date of filing *(day / month / year)*

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a) any applicant named in part 3 is not an inventor, or
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Statement of inventorship and right to grant of a patent *(Patents Form 7/77)*	
Request for preliminary examination and search *(Patents Form 9/77)*	
Request for substantive examination *(Patents Form 10/77)*	
Any other documents *(please specify)*	

11. I/We request the grant of a patent on the basis of this application.

Signature C Shepherd Date 17/7/03.

Avecia Limited Authorised Signatory

12. Name and daytime telephone number of person to contact in the United Kingdom — Mrs K M Pinder/Mrs G Shepherd 0161 721 1361/2

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COMPOSITIONS

The present invention relates to compositions comprising a particulate solid, an organic medium and a dispersant and to their use in inks, millbases, plastics and paints. Some of the dispersants are new.

Many formulations such as inks, paints, mill-bases and plastics materials require effective dispersants for uniformly distributing a particulate solid in an organic medium. The organic medium may vary from a polar to non-polar organic medium. Consequently, dispersants are sought which can disperse a particulate solid in both a polar and a non-polar organic medium.

US 4,865,621 discloses motor fuel compositions comprising the reaction product of a dibasic acid anhydride, a polyoxyalkylene monoamine and a hydrocarbyl polyamine having a number average molecular weight of up to 1343.

It has been found that certain dispersants show excellent ability to disperse a particulate solid in a range of organic media. Thus according to the present invention there is provided a composition comprising a particulate solid, an organic medium and a compound of Formula (1) and salts thereof:

$$RO\text{-}(Y)_x\text{-}T\text{-}\underset{\substack{|\\ H}}{N}\text{-}A\text{-}Z\text{-}(A'\text{-}OH)_p$$

Formula (1)

wherein:

R is C_{1-50}- optionally substituted hydrocarbyl;

Y is C_{2-4}-alkyleneoxy;

x is from 2 to 60;

T is C_{2-4}-alkylene;

Z is the residue of a polyamine and/or polyimine;

A and A^1 are independently, the residue of a dibasic acid which may be the same or different; and

p is from 0 to 200.

R is preferably aryl, aralkyl, alkaryl, cycloalkyl or alkyl, which may be linear or branched.

When R is aryl it is preferably naphthyl or phenyl.

When R is aralkyl it is preferably 2-phenylethyl or preferably benzyl.

When R is alkaryl it is preferably octyl phenyl or nonyl phenyl.

When R is cycloalkyl it is preferably C_{3-8}-cycloalkyl such as cyclopropyl and especially cyclohexyl.

It is especially preferred that R is optionally branched alkyl and especially C_{1-36} optionally branched alkyl. The group RO- may thus be the residue of an alcohol such as methanol, ethanol, n-propanol, n-butanol, n-hexanol, n-octanol, n-decanol, n-dodecanol, n-tetradecanol, n-dexadecanol, n-octodecanol, isopropanol, isobutanol, tert-butanol, 2-ethylbutanol, 2-ethylhexanol, 3-heptanol, 3,5,5-trimethylhexanol, 3,7-dimethyloctanol and the so-called Guerbat alcohols such as those which are commercially available under the trade name Isofol (ex Condea GmbH) including mixtures thereof. Specific examples of Guerbat alcohols are Isofol 12, 14T, 16, 18T, 18E, 20, 24, 28, 32, 32T and 36.

It is especially preferred that R is C_{1-6}-alkyl and especially C_{1-4}-alkyl such as methyl.

When R is substituted hydrocarbyl, the substituent may be C_{1-10}-alkoxy, carbonyl, sulphonyl, carbamoyl, sulphamoyl, halogen, nitrile, ureido, urethane or ester (i.e. -COO- or -OCO-). However, it is much preferred that R is unsubstituted.

The chain represented by $(Y)_x$ may contain only one type of C_{2-4}-alkyleneoxy repeat unit or it may contain two or more different C_{2-4}-alkyleneoxy repeat units. When the chain represented by $(Y)_x$ contains two or more different C_{2-4}-alkyleneoxy repeat units the structure of $(Y)_x$ may be random but is preferably block.

Y is preferably C_{3-4}-alkyleneoxy, more preferably $-CH_2CH_2CH_2CH_2O-$ or $-CH_2CH(CH_3)CH_2O-$ and especially $-CH_2CH(CH_3)O-$.

When Y is C_{3-4}-alkyleneoxy the chain represented by $(Y)_x$ may contain up to 95% , more preferably up to 75% and especially up to 50% by number of ethyleneoxy repeat units.

When Y is C_{3-4}-alkyleneoxy and the chain represented by $(Y)_x$ contains ethyleneoxy ($-CH_2CH_2O-$) the structure of $(Y)_x$ may be random but is preferably block.

A preferred compound of Formula (1) is wherein Y is $-CH_3CH(CH_3)O-$ and the chain represented by $(Y)_x$ may contain up to 75% by number of ethyleneoxy repeat units.

T is preferably a C_{3-4}-alkylene, more preferably T is $-CH_2CH(CH_3)-$.

Preferably T is $-CH_2CH(CH_3)-$ when Y is $-CH_2CH(CH_3)O-$.

The group RO-$(Y)_x$-T-NH- is preferably the residue of a polyalkyleneoxide monoalkyl ether monoamine. Compounds of this type are commercially available as the Jeffamine™ M-series of monoamines from Huntsman Corporation. Specific examples of Jeffamine™ amines are M-600 (9,0,600), M-1000 (3,18,1000), M-2005 (32,2,2000) and M-2070 (10, 31, 2000). The figures in parentheses are approximately repeat units of propylene oxide, ethylene oxide and number-average molecular weight respectively.

When Z is the residue of a polyamine it is preferably polyvinylamine or polyallylamine. Polyallylamine and poly(N-alky)allylamines of differing molecular weight are commercially available from Nitto Boseki. Polyvinylamine of differing molecular weight are available from Mitsubishi Kasei.

When Z is the residue of a polyimine it is preferably poly (C_{2-6}-alkyleneimine) and especially polyethyleneimine (PEI). The polyimine may be linear or especially branched.

Linear polyethyleneimine may be prepared by the hydrolysis of poly(N-acyl) alkyleneimines as described, for example, by Takeo Saegusa et al in *Macromolecules*, 1972, Vol 5, page 4470. Branched polyethyleneimines of differing molecular weight are commercially available from BASF and Nippon Shokubai. Polypropyleneimine dendrimers are commercially available from DSM Fine Chemicals and poly(amidoamine) dendrimers are available as "Starburst" dendrimers from Aldrich Chemical Company.

The number average molecular weight of the polyamine or polyimine is preferably from 500 to 600,000, more preferably from 1,500 to 200,000, even more preferably from 1,500 to 100,000 and especially from 1500 to 50,000. In the case of polyethyleneimine, the number-average molecular weight is preferably not less than 1500, more preferably not less than 3,000 and especially not less than 5,000.

The residue of dibasic acid represented by A and A^I may be derived from any dibasic acid of formula HOOC-B-COOH or anhydride thereof wherein B is a direct bond or a divalent organic moiety containing from 1 to 20 carbon atoms. B may be aromatic, hetero aromatic, alicyclic or aliphatic which may be optionally substituted. When B is aliphatic containing two or more carbon atoms, it may be linear or branched, saturated or unsaturated. Preferably B is unsubstituted. It is also preferred that B contains not greater than 12 and especially not greater than 8 carbon atoms.

When B is aromatic it is preferably phenylene, when B is alicyclic it is preferably cyclohexylene and when B is aliphatic it is preferably alkylene. Preferred dibasic acids are terephthalic, tetrahydrophthalic, methyl tetrahydrophthalic, hexahydrophthalic, methyl hexahydrophthalic, trimellitic, C_{1-20}-alkenyl or alkyl succinic and especially maleic, malonic, succinic and phthalic acids. Preferred anhydrides are glutaric, succinic and phthalic anhydrides.

Mixtures of dibasic acids or anhydrides may be used.

When a mixture of two or more acids or anhydrides are used for A the result is a mixture of compounds of Formula (1) having different acid or anhydride residues as A. Preferably only one type of acid or anhydride is used for A.

When a mixture of two or more acids or anhydrides is used for A^I the result is a mixture of compounds of Formula (1) where different compounds have different amounts and positioning on Z of the residues of the acids or anhydrides. One type of acid or anhydride or a mixture of two of more acids or anhydrides can be used for A^I.

A preferred mixture of acids or anhydrides for A^I contains succinic acid or anhydride and C_{1-20}-alkyl succinic acid or anhydride.

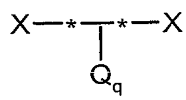
When a mixture of acids or anhydrides is used for A^I it is preferable that one of these acids or anhydrides is used alone for A.

When one acid or anhydride is used for A and one is used for A^I then preferably A and A^I are the same.

When p is other than zero it is preferred that the majority of amine/imine groups in Z which do not carry the group RO-$(Y)_x$-T-NH-A- are connected to the residue

– A^{I} -OH.

The polyamine or polyimine represented by Z preferably carries 2 or more groups RO-$(Y)_x$-T-NH-A- which may be the same or different. Dispersants of this type may be conveniently represented by Formula (2):



Formula (2)

wherein

X-*-*-X represents the polyamine and/or polyimine;

Q is the chain RO-$(Y)_x$-T-NH-A-; and

q is from 2 to 2000.

As noted hereinbefore the dispersant may be present in the form of a salt. The salt may be that of an alkali metal such as lithium, potassium or sodium. Alternatively the salt may be formed with ammonium, amine or quaternary ammonium cation. Examples of amines are methylamine, diethylamine, ethanolamine, diethanolaime, hexylamine, 2-ethylhexylamine and octadecylamine. The quaternary ammonium cation may be a quaternary ammonium cation or a benzalkonium cation. The quaternary ammonium cation preferably contains one or two alkyl groups containing from 6 to 20 carbon atoms. Examples of quaternary ammonium cations are tetraethyl ammonium, N-octadecyl-N,N,N-trimethyl ammonium; N,N-didocyl-N,N-dimethyl ammonium, N-benzyl-N,N,N-trimethyl ammonium and N-benzyl-N-octadecyl.

When p is other than zero it is much preferred that the dispersant is in the form of a free acid.

When p is zero the dispersant may be in the form of a salt of a coloured acid. The coloured acid may be a sulphonated phthalocyanine, especially a copper or nickel phthalocyanine or a disazo dyestuff containing a sulphonic acid and/or carboxylic acid group.

When p is zero some of the amine/imine groups in Z which do not carry the group RO-$(Y)_x$-T-NH-A- can be quaternised. Preferred quaternisation agents are dimethyl sulphate, benzyl chloride, methyl halides especially Cl, Br and I, methyl p-toluenesulfonate and propane sultone.

The compound of Formula (1) may be made by any method known to the art. It is preferably prepared by the reaction of a compound of Formula (3) with a dibasic acid or more preferably anhydride thereof and a polyamine and/or a polyimine and optionally a second dibasic acid or preferably anhydride thereof.

$$RO\text{-}(Y)_x\text{-}T\text{-}NH_2$$

Formula (3)

wherein R, Y, T and x are as described hereinbefore.

Preferably the compound of Formula (3) is reacted with the first dibasic acid or anhydride at a temperature from 40 to 150°C, more preferably from 60 to 100°C. The reaction is preferably performed in an inert atmosphere. The inert atmosphere may be provided by any inert gas of the Periodic Table but is preferably nitrogen.

The reaction with the polyamine and/or polyimine is preferably carried out at a temperature of from 100 to 150°C. Under such conditions the reaction results in a mixture of amide and salt forms rather than the salt form alone.

The reaction involving the optional second dibasic acid or preferably anhydride thereof is preferably carried out using similar conditions to those employed using the first dibasic acid or anhydride thereof

The reaction involving the first and the second dibasic acid or anhydride thereof may be carried out in the presence of an organic diluent which is inert to the reactants. Preferably, the organic diluent is a solvent for the reactants. The organic diluent may be aromatic or aliphatic including halogenated derivatives. Examples are toluene, chlorobenzene, heptane and petroleum ether distillates. Preferably the reaction is carried out in the absence of an organic diluent.

The particulate solid present in the composition may be any inorganic or organic solid material which is substantially insoluble in the organic medium. Preferably the particulate solid is a pigment.

Examples of suitable solids are pigments for solvent inks; pigments, extenders and fillers for paints and plastics materials; disperse dyes; optical brightening agents and textile auxiliaries for solvent dyebaths, inks and other solvent application systems; solids for oil-based and inverse-emulsion drilling muds; dirt and solid particles in dry cleaning fluids; particulate ceramic materials; magnetic materials and magnetic recording media; fibres such as glass, steel, carbon and boron for composite materials, and biocides, agrochemicals and pharmaceuticals which are applied as dispersions in organic media.

A preferred solid is an organic pigment from any of the recognised classes of pigments described, for example, in the Third Edition of the Colour Index (1971) and subsequent revisions of, and supplements thereto, under the chapter headed "Pigments". Examples of organic pigments are those from the azo, disazo, condensed azo, thioindigo, indanthrone, isoindanthrone, anthanthrone, anthraquinone, isodibenzanthrone, triphendioxazine, quinacridone and phthalocyanine series, especially copper phthalocyanine and its nuclear halogenated derivatives, and also lakes of acid, basic and mordant dyes. Carbon black, although strictly inorganic, behaves more like an organic

pigment in its dispersing properties. Preferred organic pigments are phthalocyanines, especially copper phthalocyanines, monoazos, disazos, indanthrones, anthranthrones, quinacridones and carbon blacks.

Preferred inorganic solids include: extenders and fillers such as talc, kaolin, silica, barytes and chalk; particulate ceramic materials such as alumina, silica, zirconia, titania, silicon nitride, boron nitride, silicon carbide, boron carbide, mixed silicon-aluminium nitrides and metal titanates; particulate magnetic materials such as the magnetic oxides of transition metals, especially iron and chromium, e.g. gamma-Fe_2O_3, Fe_3O_4, and cobalt-doped iron oxides, calcium oxide, ferrites, especially barium ferrites; and metal particles, especially metallic iron, nickel, cobalt, copper and alloys thereof.

Other useful solid materials include agrochemicals such as the fungicides flutriafen, carbendazim, chlorothalonil and mancozeb.

The organic medium present in the composition of the invention is preferably a plastics material, more preferably an organic liquid. The organic liquid may be a non-polar or more preferably a polar organic liquid. By the term "polar" in relation to the organic liquid it is meant that an organic liquid is capable of forming moderate to strong bonds as described in the article entitled "A Three Dimensional Approach to Solubility" by Crowley et al in Journal of Paint Technology, Vol. 38, 1966, at page 269. Such organic liquids generally have a hydrogen bonding number of 5 or more as defined in the abovementioned article.

Examples of suitable polar organic liquids are amines, ethers, especially lower alkyl ethers, organic acids, esters, ketones, glycols, alcohols and amides. Numerous specific examples of such moderately strongly hydrogen bonding liquids are given in the book entitled "Compatibility and Solubility" by Ibert Mellan (published in 1968 by Noyes Development Corporation) in Table 2.14 on pages 39-40 and these liquids all fall within the scope of the term polar organic liquid as used herein.

Preferred polar organic liquids are dialkyl ketones, alkyl esters of alkane carboxylic acids and alkanols, especially such liquids containing up to, and including, a total of 6 carbon atoms. As examples of the preferred and especially preferred liquids there may be mentioned dialkyl and cycloalkyl ketones, such as acetone, methyl ethyl ketone, diethyl ketone, di-isopropyl ketone, methyl isobutyl ketone, di-isobutyl ketone, methyl isoamyl ketone, methyl n-amyl ketone and cyclohexanone; alkyl esters such as methyl acetate, ethyl acetate, isopropyl acetate, butyl acetate, ethyl formate, methyl propionate, methoxy propylacetate and ethyl butyrate; glycols and glycol esters and ethers, such as ethylene glycol, 2-ethoxyethanol, 3-methoxypropylpropanol, 3-ethoxypropylpropanol, 2-butoxyethyl acetate, 3-methoxypropyl acetate, 3-ethoxypropyl acetate and 2-ethoxyethyl acetate; alkanols such as methanol, ethanol, n-propanol, isopropanol, n-butanol and isobutanol and dialkyl and cyclic ethers such as diethyl ether and tetrahydrofuran. Especially preferred solvents are alkanols, alkane carboxylic acids and esters of alkane carboxlic acids.

Examples of organic liquids, which can be used as polar organic liquid are film-forming resins such as are suitable for the preparation of inks, paints and chips for use in various applications such as paints and inks. Examples of such resins include polyamides, such as Versamid™ and Wolfamid™, and cellulose ethers, such as ethyl cellulose and ethyl hydroxyethyl cellulose, nitrocellulose and cellulose acetate butyrate resins, including mixtures thereof. Examples of paint resins include short oil alkyd/melamine-formaldehyde, polyester/melamine-formaldehyde, thermosetting acrylic/melamine-formaldehyde, long oil alkyd, polyether polyols and multi-media resins such as acrylic and urea/aldehyde.

The organic liquid may be a polyol, that is to say, an organic liquid with two or more hydroxy groups. Preferred polyols include alpha–omega diols, especially alpha–omega diol ethoxylates.

Preferred non-polar organic liquids are compounds containing aliphatic groups, aromatic groups or mixtures thereof.

Preferred non-polar organic liquids are non-halogenated aromatic hydrocarbons (e.g. toluene and xylene), halogenated aromatic hydrocarbons (e.g. chlorobenzene, dichlorobenzene, chlorotoluene), non-halogenated aliphatic hydrocarbons (e.g. linear and branched aliphatic hydrocarbons containing six or more carbon atoms both fully and partially saturated), halogenated aliphatic hydrocarbons (e.g. dichloromethane, carbon tetrachloride, chloroform, trichloroethane) and natural non-polar organics (e.g. vegetable oil, sunflower oil, linseed oil, terpenes and glycerides).

Preferably the organic liquid comprises at least 0.1% by weight, more preferably 1% by weight of a polar organic liquid based on the total organic liquid.

The organic liquid may further comprise water.

When the organic liquid contains water it is preferably not greater than 70%, more preferably not greater than 50%, especially not greater than 40% by weight based on the amount of organic liquid.

The plastics material may be a thermoset resin or a thermoplastic resin. The thermosetting resins useful in this invention include resins which undergo a chemical reaction when heated, catalysed, or subject to UV radiation and become relatively infusible. Typical reactions in thermosetting resins include oxidation or unsaturated double bonds, reactions involving epoxy/amine, epoxy/carbonyl, epoxy/hydroxyl, polyisocyanate/hydroxy, amino resin/hydroxy moieties, free radical reactions or polyacrylate, cationic polymerization or epoxy resins and vinyl ether, condensation or silanol, etc.

Polymers with hydroxy functionality (frequently polyols) are widely used in thermosetting system to crosslink with amino resins or polyisocyanates. The polyols include acrylic polyols, alkyd polyols, polyester polyols, polyether polyols and polyurethane polyols. Typical amino resins include melamine formaldehyde resins, benzoguanamine formaldehyde resins, urea formaldehyde resins and glycoluril

formaldehyde resins. Polyisocyanates are resins with two or more isocyanate groups, including both monomeric aliphatic diisocyanates, monomeric aromatic diisocyanates and their polymers. Typical aliphatic diisocyanates include hexamethylene diisocyanate, isophorone diisocyanate and hydrogenated diphenylmethane diisocyanate. Typical aromatic isocyanates include toluene diisocyanates and biphneylmethane diisocyanates.

Particularly preferable thermoplastic resins include polyolefins, polyesters, polyamides, polycarbonates, polyurethanes, polystyrenics, poly(meth)acrylates, celluloses and cellulose derivatives. Said compositions can be prepared in a number of ways but melt mixing and dry solid blending are typical methods.

If desired, the compositions may contain other ingredients, for example resins (where these do not already constitute the organic medium), binders, fluidising agents anti-sedimentation agents, plasticisers, surfactants, anti-foamers, rheology modifiers, levelling agents, gloss modifiers and preservatives.

The compositions typically contain from 1 to 95% by weight of the particulate solid, the precise quantity depending on the nature of the solid and the quantity depending on the nature of the solid and the relative densities of the solid and the polar organic liquid. For example, a composition in which the solid is an organic material, such as an organic pigment, preferably contains from 15 to 60% by weight of the solid whereas a composition in which the solid is an inorganic material, such as an inorganic pigment, filler or extender, preferably contains from 40 to 90% by weight of the solid based on the total weight of composition.

The composition may be prepared by any of the conventional methods known for preparing dispersions. Thus, the solid, the organic medium and the dispersant may be mixed in any order, the mixture then being subjected to a mechanical treatment to reduce the particles of the solid to an appropriate size, for example by ball milling, bead milling, gravel milling or plastic milling until the dispersion is formed. Alternatively, the solid may be treated to reduce its particle size independently or in admixture with either the organic medium or the dispersant, the other ingredient or ingredients then being added and the mixture being agitated to provide the composition.

The composition of the present invention is particularly suited to liquid dispersions. For such dispersions a preferred composition comprises:

a) from 0.5 to 30 parts of a particulate solid;

b) from 0.5 to 30 parts of a compound of Formula (1); and

c) from 40 to 99 parts of an organic liquid;

wherein all parts are by weight and the amounts a) +b) +c) = 100.

More preferably component a) comprises from 0.5 to 30 parts of a pigment and such dispersions are useful as liquid inks, paints and mill-bases.

If a composition is required comprising a particulate solid and a dispersant of Formula (1) in dry form, the organic liquid is preferably volatile so that it may be readily

Thus according to a further aspect of the present invention there is provided a paint or ink comprising a particulate solid, an organic liquid, a binder and a compound of Formula (1) and salts thereof.

As noted hereinbefore, many of the dispersants of Formula (1) are novel.

Thus, according to a further aspect of the invention there is provided a compound of Formula (4) and salts thereof.

$$\mathrm{RO\text{-}(Y)_x\text{-}T\text{-}\underset{\underset{H}{|}}{N}\text{-}A\text{-}Z\text{-}(A'\text{-}OH)_r}$$

Formula (4)

wherein R, Y, T, A, Z, A^I, x, $(Y)_x$ and the salts are as defined hereinbefore and r is from 1 to 200.

A more preferred compound of Formula (4) and salts thereof is provided wherein R, T, A, Z, A^I, x, the salts and r are as defined hereinbefore, Y is $C_{3\text{-}4}$-alkyleneoxy and the chain represented by $(Y)_x$ may contain up to 75% by number of ethyleneoxy repeat units.

According to a still further aspect of the invention there is provided a compound of Formula (1) and salts thereof wherein Z is a polyamine and/or polyimine having a number average molecular weight of not less than 1500.

A more preferred compound of Formula (1) and salts thereof is provided wherein Y is $C_{3\text{-}4}$-alkyleneoxy, the chain represented by $(Y)_x$ may contain up to 75% by number of ethyleneoxy repeat units and Z is a polyamine and/or polyimine having a number average molecular weight of not less than 1500.

The invention is further illustrated by the following examples wherein all references to amounts are in parts by weight unless indicated to the contrary.

Examples

Example 1 M2005 SA (1:1) PEI (13:1)

Succinic anhydride (2.5 parts, 25mmols supplied from Aldrich) was added to stirred Jeffamine™ M2005 (50 parts, 25mmols supplied from Huntsman) under a nitrogen gas atmosphere. The temperature was raised to 80°C and the mixture stirred continuously for a duration of 8 hrs. Infra red spectroscopy of the mixture showed that some anhydride groups remained. 1.7 parts of Jeffamine™ M2005 was then added to the stirred mixture which was further reacted at 80°C for 1hr. Infra red spectroscopy of this mixture showed that all of the anhydride groups had now been successfully reacted. The product was obtained as a pale yellow viscous oil (53.5g). This is Intermediate 1.

Intermediate 1 (53.5 parts) was stirred with polyethyleneimine SP200 (4.1g, ex Nippon Shokubia) and heated to a temperature of 120°C for a duration of 6 hrs under a nitrogen gas atmosphere. After cooling to 25°C the product was obtained as an amber viscous liquid (55 parts) wherein the weight ratio of the polyether chain to PEI is 13:1. This is Dispersant 1.

Example 2 M2005 SA (1:1) PEI (17:1)

Example 1 was repeated except that the amount of polyethyleneimine was reduced to 3.15 parts. The product was obtained as an amber viscous liquid (55 parts) where the weight ratio of the polyether chain to PEI is 17:1. This is Dispersant 2.

Example 3 M600 SA (1:1) PEI (6:1)

Succinic anhydride (8.34 parts, 83.3mmols) was added to stirred Jeffamine™ M600 (50 parts, 83.3mmols supplied from Huntsman) under a nitrogen gas atmosphere. The temperature was raised to 80°C and the mixture was stirred continuously for a duration of 6 hrs. Infra red spectroscopy of this mixture showed that all of the anhydride groups had been successfully reacted. This is Intermediate 2.

Intermediate 2 (24 parts) was stirred with polyethyleneimine SP200 (4.0 parts) at a temperature of 80°C under a nitrogen atmosphere. The mixture was heated to 120°C and stirred for 6 hours still under a nitrogen atmosphere. After cooling to 25°C the product was obtained as a brown viscous liquid/gum (26 parts) where the weight ratio of polyether chain to PEI is 6:1. This is Dispersant 3.

Example 4 M2005 M600 SA (8.5:3.5:1) PEI (10:1)

Succinic anhydride (2.95 parts, 29.5mmols) was added to a stirred mixture of Jeffamine™ M600 (10.2 parts, 17mmols) and Jeffamine™ M2005 (25 parts, 12.5mmols) under a nitrogen atmosphere. The temperature was raised to 80°C and the mixture was stirred for a duration of 6 hrs. Infra red spectroscopy showed that all of the anhydride groups had been successfully reacted. After cooling to 25°C the product was obtained as a yellow viscous liquid. This is Intermediate 3.

Polyethyleneimine SP200 (3.0 parts) was added to Intermediate 3 (30.0 parts) at a temperature of 80°C. The temperature was raised to 120°C and the mixture was stirred for a duration of 6 hrs under a nitrogen atmosphere. After cooling to 25°C the product was obtained as an amber viscous liquid (40 parts), wherein the weight ratio of M2005 to M600 to succinic acid is 8.5:3.5:1 and the weight ratio of the polyether chain to PEI is 10:1. This is Dispersant 4.

Example 5 M2005 M600 SA (14.3:1.75:1) PEI (13:1)

Jeffamine M600 (6.13 parts, 10.2mmols), Jeffamine M2005 (50 parts, 25 mmols) and succinic anhydride (3.5 parts, 35mmols) were stirred at 80°C under nitrogen for 6 hours. After cooling to 25°C the product was obtained as a yellow viscous liquid. This is Intermediate 4.

Polyethyleneimine SP200 (3.0 parts) was added to Intermediate 4 (39 parts) at 80°C. The reactants were stirred under nitrogen at 120°C for 6 hours. After cooling to 25°C, the product was obtained as an amber viscous liquid (40 parts) wherein the weight ratio of polyether chain to PEI is 13:1. This is Dispersant 5.

Preparation of Mill-bases

A series of magenta mill-bases were prepared utilising Dispersants 1 to 5. The mill-bases were prepared by dissolving the dispersant (0.40 parts) in the solvent indicated in Table 1. Glass beads (3mm, 17 parts) and Monolite Rubine 3B (ex Heubach 2.0 parts) were added and the mixture was shaken on a horizontal shaker for 16 hours. The resulting dispersions were then assessed for fluidity using an arbitrary scale of A to E (good to bad). The results are given in Table 1.

TABLE 1

Dispersant	**Toluene**	**4:1 MPA : Butanol**	**4:1 Butanol : MPA**	**Ethanol**
1	A/B	A	A	A
2	B	A/B	A	A
3	C/D	C	C	C
4	B	A/B	A/B	A
5	B	A/B	A/B	A
Control	A/B	B/C	D/E	E

Footnote to Table 1:

The control is poly(ε-caprolactone) reacted with polyethyleneimine as described in US 4,645,611.

MPA is methoxypropylacetate.

Table 1 shows that dispersants of the present invention give good fluidity with organic media of very different polarities.

CLAIMS

1. A composition comprising a particulate solid, an organic medium and a compound of Formula (1) and salts thereof:

$$RO\text{-}(Y)_x\text{-}T\text{-}\underset{\displaystyle H}{\underset{|}{N}}\text{-}A\text{-}Z\text{-}(A'\text{-}OH)_p$$

Formula (1)

wherein:

- R is C_{1-50}- optionally substituted hydrocarbyl;
- Y is C_{2-4}-alkyleneoxy;
- x is from 2 to 60;
- T is C_{2-4}-alkylene;
- Z is the residue from a polyamine and/or polyimine;
- A and A^I are independently, the residue of a dibasic acid which may be the same or different; and
- p is from 0 to 200.

2. A composition according to claim 1 wherein Y is C_{3-4}-alkyleneoxy and the chain represented by $(Y)_x$ may contain up to 75% by number of ethyleneoxy repeat units.

3. A composition according to any one of the preceding claims wherein A and A^I are the residues independently derived from the group consisting of maleic acid, malonic acid, succinic and phthalic acid.

4. A composition according to any one of the preceding claims wherein the group represented by Z is polyethyleneimine.

5. A composition according to any one of the preceding claims wherein the organic medium is an organic liquid.

6. A composition according to any one of claims 1 to 4 wherein the organic medium is a plastics material.

7. A composition according to claim 5 wherein the organic liquid comprises at least 0.1% by weight of a polar organic liquid based on the total organic liquid.

8. A composition according to any one of the preceding claims wherein the particulate solid is a pigment.

9. A mill-base comprising a particulate solid, an organic liquid and a compound of Formula (1) and salts thereof:

$$RO\text{-}(Y)_x\text{-}T\text{-}\underset{\displaystyle H}{\underset{|}{N}}\text{-}A\text{-}Z\text{-}(A'\text{-}OH)_p$$

Formula (1)

wherein:

R is C_{1-50}- optionally substituted alkyl;
Y is C_{2-4}-alkyleneoxy;
x is from 2 to 60;
T is C_{2-4}-alkylene;
Z is the residue from a polyamine and/or polyimine;
A and A^{I} are independently, the residue of a dibasic acid which may be the same or different; and
p is from 0 to 200.

10. A paint or ink comprising a particulate solid, an organic liquid, a binder and a compound of Formula (1) and salts thereof:

$$RO\text{-}(Y)_x\text{-}T\text{-}\underset{\displaystyle H}{\underset{|}{N}}\text{-}A\text{-}Z\text{-}(A'\text{-}OH)_p$$

Formula (1)

wherein:

R is C_{1-50}- optionally substituted alkyl;
Y is C_{2-4}-alkyleneoxy
x is from 2 to 60;
T is C_{2-4}-alkylene;
Z is the residue from a polyamine and/or polyimine;
A and A^{I} are independently, the residue of a dibasic acid which may be the same or different; and
p is from 0 to 200.

11. A compound of Formula (4) and salts thereof:

$$\mathrm{RO\text{-}(Y)_x\text{-}T\text{-}\underset{\displaystyle H}{\underset{|}{N}}\text{-}A\text{-}Z\text{-}(A'\text{-}OH)_r}$$

Formula (4)

wherein:

R is C_{1-50}- optionally substituted alkyl;

Y is C_{2-4}-alkyleneoxy;

x is from 2 to 60;

T is C_{2-4}-alkylene;

Z is the residue of a polyamine and/or polyimine;

A and A^{I} are independently, the residue of a dibasic acid which may be the same or different; and

r is from 1 to 200.

12. A compound of Formula (5) and salts thereof:

$$\mathrm{RO\text{-}(Y)_x\text{-}T\text{-}\underset{\displaystyle H}{\underset{|}{N}}\text{-}A\text{-}Z\text{-}(A'\text{-}OH)_p}$$

Formula (5)

wherein:

R is C_{1-50}- optionally substituted alkyl;

Y is C_{2-4}-alkyleneoxy;

x is from 2 to 60;

T is C_{2-4}-alkylene;

Z is the residue of a polyamine and/or polyimine having a number average molecular weight of not less than 1,500;

A and A^{I} are independently, the residue of a dibasic acid which may be the same or different; and

p is from 0 to 200.